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(54) PREPARATION OF CHEMICAL PULP AND XYLOSE, UTILIZING A DIRECT ACID HYDROLYSIS ON THE PULP

HERSTELLUNG VON CHEMISCHEN ZELLSTOFF UND XYLOSE UNTER ANWENDUNG DIREKTER SAURER HYDROLYSE DES ZELLSTOFFES

PREPARATION DE PATE A PAPIER CHIMIQUE ET DE XYLOSE PAR HYDROLYSE ACIDE REALISEE DIRECTEMENT SUR LA PATE A PAPIER

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- **DATABASE CAPLUS [Online] KRAMAR ALOJZ ET AL.: 'Pentoses from beech bark', XP002954012 Retrieved from STN International, accession no. 93:150586 Database accession no. 1980:550586 & CS 181 485 B 15 January 1980**

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Description

BACKGROUND OF THE INVENTION

[0001] The invention relates to the preparation of chemical pulp and xylose, and particularly to recovering xylose from pulp, such as sulphate pulp, prepared by alkaline or neutral cooking, and simultaneously achieving the desired characteristics for the pulp. The method of the invention utilizes a direct acid hydrolysis of the pulp, resulting in a good xylose yield. At the same time, the obtained pulp is usable as paper pulp or dissolving pulp.

[0002] In a plurality of plants, the main portion of hemicellulose is xylan, which can be hydrolyzed to xylose. The foremost starting material for xylan is hemicellulose from hardwood, particularly birch, mainly composed of xylan.

[0003] For example birch sulphate pulp typically contains about 15 to 25% xylan, which is usable as a raw material of xylose. When xylose is prepared from pulp, the problem involved has been to achieve sufficient xylose yields and to simultaneously achieve acceptable characteristics for the pulp.

[0004] Finnish Patent 55516 (Kemi Oy) discloses a method of preparing pure xylan, suitable for the raw material of xylose and xylitol, in particular. The method uses bleached or unbleached hardwood cellulose as the raw material. The cellulose is treated with an alkali solution, whereby the hemicelluloses are dissolved. The alkali solution containing hemicellulose is pressed and filtered from pulp. The dissolved hemicellulose is precipitated by the addition of carbon dioxide to the solution, whereby the xylan precipitates. In the method, most of the xylan in the pulp, the xylan being in principle usable as the raw material of xylose, is, however, not utilized. Moreover, the method uses much alkali.

[0005] Several methods are also known in which enzymatic hydrolysis is used to separate hemicellulose components from the pulp. For example Paice, M.G. & Jurasek, L., *Removing Hemicellulose from Pulps by Specific Enzymic Hydrolysis*, J. Wood Chem, and Tech., 4 (2), 187 to 198, 1984, describes a method of separating hemicellulose from aspen pulp by xylanase treatment. The most important hemicellulose products thus obtained were xylan and xylobiose. However, the enzyme dose is uneconomically large.

[0006] WO 98/56958 (Xyrofin Oy) discloses a method of preparing xylose by first extracting xylan from a cellulose pulp or its alkali solution with an aqueous solution of a xylanase enzyme and by then using acid to hydrolyze the xylan in the solution to xylose. However, the acid hydrolysis is not performed directly on the chemical pulp, and therefore all the xylan in the pulp cannot be utilized.

[0007] Acid hydrolysis is also applied as pre-hydrolysis in the separation of xylose from wood material. In this case, the acid hydrolysis is performed directly on wood chips before the pulp is prepared. One such method is disclosed in Guangyu, Yao et al., *Production of Pulp and Recovery of Xylose from Hardwood. II. The Optimal Process Conditions for Prehydrolysis of Eucalyptu Citriodora Chips with Dilute Sulphuric Acid and Sulfate Pulping,* Journal of Nanking Technological College of Forest Products, No. 4 (1988), p. 32. The publication relates to a method of preparing sulphate pulp with simultaneous recovery of xylose. As raw material is used Eucalyptu Citriodora chips, on which prehydrolysis is carried out with dilute sulphuric acid. A xylose-containing solution and prehydrolyzed wood chips are obtained. Sulphate pulp is then prepared from the prehydrolyzed wood chips. The pulp is said to be suitable for the preparation of viscose, for example.

[0008] US Patent 4,008,285 (Melaja, A. J. & Hämäläinen, L.) discloses a method of recovering xylitol from a xylan-containing raw material, which may be for example wood material, such as birch chips. The birch chips are first hydrolyzed with for example acid, the hydrolysate is purified and the purified hydrolysate is subjected to chromatographic fractionation to provide a solution containing a high level of xylose. However, the pulp is not recovered in this method.

[0009] STN International , Database Caplus, caplus no. 1980:550586, Kramar Alojz et al.: "Pentoses from beech bark"; & Czech. SC 181485 19800115, 3 pp. discloses heating pulped bark containing 17% pentosans with 1% H_2SO_4 for six hours at 100 °C in an autoclave, followed by working up and concentrating the hydrolysate to give a 5.9% syrup (based on the dry weight of the bark) containing 90% L-arabinose. Analogous hydrolysis of pulped bark with 2% H_2SO_4 at 120°C and work-up gave a product containing 80% xylose.

[0010] US Patent 4,734,162 (The Procter & Gamble Company) disloses a pulp produced by a process comprising the steps of providing hardwood chips, introducing said chips into a digester, providing a cooking liquor comprising from about 0.4% to about 3% ammonia and from about 9% to about 14% sulfur dioxide, sulfonating the lignin in said chips at less than about 110°C, and hydrolyzing the sulfonated lignin at a temperature of about 140°C to about 155°C at a pH of from about 2 to about 3. It is recited that the pulp thus obtained comprises hardwood cellulosic fibers and has a xylan content of between about 6% to about 8% by weight on a dry pulp basis. It is also recited that the pulp thus obtained can be made into useful tissue paper webs having enhanced softness properties.

[0011] US Patent 3,954,497 (Friese, H. , Süd-Chemie A.G.) discloses a process for the hydrolysis of deciduous wood, wherein the hydrolysis is carried out in a first stage with an alkali metal hydroxide solution having a concentration of less than 4 percent by weight, and in a second stage with a mineral acid to provide a solid residue containing lignin and an acidic solution of D-(+)-xylose. The solid residue containing lignin is separated from the acidic solution of D-(+)-xylose, the residue is extracted with organic solvent to remove the lignin therefrom and the remaining residue is treated

at an elevated temperature with alkali metal chlorite, followed by treatment with alkali metal hydroxide solution to provide cellulose.

[0012] US Patent 4,070,232 (Harald F. Funk) discloses prehydrolysis of plant material containing hemicellulose and lignin in the presence of steam and vapours of a dilute acid solution at a pH of 1.5 to 3.5 at a temperature between about 105°C and about 135°C to hydrolyze the hemicellulose into pentoses and hexoses and to leave a fibrous material and a liquid, said liquid containing pentoses and hexoses. The liquid is separated from the fibrous material, and the pentoses and hexoses are recovered from the liquid. The fibrous material is digested in the presence of white liquor at a temperature in the range of about 105°C to about 135°C, followed by the separation of the digested fibrous material from the spent digestion liquor.

BRIEF DESCRIPTION OF THE INVENTION

[0013] It is therefore an object of the invention to provide a method of recovering xylose from pulp prepared by alkaline or neutral cooking with a sufficient xylose yield, and, simultaneously, preparing paper pulp or dissolving pulp so as to obtain acceptable characteristics for the pulp. The objects of the invention are achieved by a method, which is characterized in what is disclosed in the independent claims. The preferred embodiments of the invention are disclosed in the dependent claims.

[0014] In accordance with the present invention, it has now been surprisingly found that high-quality paper pulp and dissolving pulp can be prepared by first subjecting the pulp to alkaline or neutral cooking and then, as post hydrolysis, to acid hydrolysis in order to recover the xylose. In the method of the invention, simultaneous extraction and hydrolysis of xylan are achieved, and extensive use of alkali in the extraction of xylan can be totally avoided.

[0015] In the context of the present invention, the expression 'sufficient xylose yield' refers to a xylose yield of at least 5% (50 g xylose/1,000 g pulp), preferably at least 10% (100 g xylose/1,000 g pulp), calculated on the dry substance of the pulp.

[0016] In the context of the present invention, the expression 'acceptable pulp characteristics' means that the viscosity of the acid-treated pulp remains sufficient for paper pulp or dissolving pulp. Typically, the viscosity of paper pulp or dissolving pulp should be at least 300 ml/g, preferably at least 450 ml/g, and most preferably at least 600 ml/g. The acceptable viscosity values depend on the final purpose of use of the pulp. If the pulp is used for the preparation of paper whose strength characteristics have to be good, a higher viscosity is required for the pulp, typically at least 600 ml/g. Pulp having a lower viscosity is feasible particularly when acid-treated pulp obtained by the method of the invention is used in a mixture with non-acid-treated pulp in the production of paper, for example.

[0017] The expression 'post hydrolysis is performed directly on the pulp' means that the acid treatment for hydrolyzing xylan to xylose is performed on the pulp itself, not for example on a xylan solution extracted from the pulp (such as in the method of WO 98/56958, for example). In this case, xylan is hydrolyzed into xylose in connection with the acid treatment of the pulp.

DETAILED DESCRIPTION OF THE INVENTION

[0018] The present invention relates to a method of preparing paper pulp or dissolving pulp and a xylose solution by the use of alkaline or neutral cooking and post hydrolysis of the pulp. The method is characterized in that the post hydrolysis is performed directly on the pulp by the use of an acid.

[0019] In the method of the invention, the pulp is typically post-hydrolyzed with an acid until the xylose yield is at least 5%, preferably at least 10%, while the viscosity of the pulp remains at a value of at least 300 ml/g, preferably 450 ml/g.

[0020] When the hydrolysis of xylan is performed directly on the pulp in connection with the acid treatment of the pulp without pre-treatment steps, the xylan contained by the pulp can be utilized as completely as possible. In this case the xylan can be hydrolyzed as efficiently as possible into xylose, and the xylose yields can be optimized.

[0021] In the present invention, alkali cooking refers to a sulphate process, soda process, soda/anthraquinone process and alkali sulphite process, and neutral cooking to a neutral sulphite process. Post hydrolysis is thus typically performed on pulp prepared by alkaline or neutral cooking, typically the sulphate process, the soda process, the soda/anthraquinone process, the alkali sulphite process or the neutral sulphite process. Sulphate pulp is the most preferably used.

[0022] In the cooking, hardwood or herbaceous plants can be used as the raw material. Examples of usable hardwood include birch, aspen, alder, oak, poplar, beech, gum tree and acacia tree. Especially important are birch and beech, for example. Examples of herbaceous plants include reed, reed canary grass, bagasse, bamboo and straw, such as corn straw.

[0023] An especially preferable raw material is birch, whereby acid hydrolysis is performed on birch sulphate pulp.

[0024] In the cooking, the pulp is cooked to the desired kappa number, which is typically in the range between 20

and 40. After the cooking, the pulp may be further subjected to oxygen delignification (to a kappa number of about 10, for example) and bleaching (typically to a kappa number of about 0).

[0025] The acid hydrolysis can be performed immediately after the cooking, oxygen delignification or bleaching (ECF bleaching, for example).

[0026] In order to make the xylan hydrolyze as efficiently as possible to xylose, the pulps can be subjected to liquor exchange before the acid treatment, whereby the solution affecting the pulp is as close as possible to the adjusted concentration. In liquor exchange, the aqueous solution contained by the pulp is replaced by the acid to be used, for example by concentrated formic acid. The liquor exchange can also be carried out by evaporating the water contained by the pulp and replacing it with the acid to be used in the acid treatment.

[0027] In the acid treatment, xylose is preferably recovered in monomer form. However, xylose can also be recovered in oligomer form.

[0028] The acid treatment can be carried out with a mineral acid or an organic acid.

[0029] The acid treatment is preferably carried out with formic acid. The concentration of the formic acid solution is typically in the range between 50 and 100%, preferably between 75 and 90%. The treatment temperature is typically between 90 and 130°C, preferably between 100 and 120°C. The duration of the formic acid treatment is typically from 15 min to 4 h, preferably from 20 min to 1.5 h.

[0030] The acid treatment can also be carried out with for example a bisulphite solution, which is usually readily available in sulphate pulp processes. In the present invention, a bisulphite solution refers to a partly neutralized aqueous solution of sulphur dioxide (SO_2) containing bisulphite ions. The SO_2 content of the bisulphite solution is typically in the range of about 1 to 5%, preferably about 3%, of which the amount of bound SO_2 is typically about 10%. When a bisulphite solution is used, the acid treatment temperature is typically about 110 to 150°C, preferably about 125 to 145°C. The duration of the bisulphite treatment is typically about 1 to 3 h.

[0031] Other usable acids include sulphuric acid, sulphurous acid and hydrochloric acid.

[0032] The treatment with formic acid is preferably carried out after bleaching (for example ECF bleaching), but it also can be carried out after oxygen delignification, or even immediately after cooking, on unbleached pulp.

[0033] The treatment with bisulphite solution is preferably carried out before oxygen delignification/bleaching, but can also be carried out after oxygen delignification or bleaching, for example ECF bleaching.

[0034] After the acid treatment, the pulp is typically washed to recover the xylose as completely as possible from the acid-treated pulp.

[0035] After the acid treatment, the obtained xylose solution and the pulp are separated, typically by filtration. The acid used, for example formic acid, is then separated from the xylose solution, typically by distillation. The separated acid is recycled and reused in hydrolysis.

[0036] The obtained xylose solution is usable for the preparation of xylose. From the xylose, xylitol can be further prepared by catalytic reduction, for example.

[0037] The xylose is usable as such, for example as a flavour and an aroma intensifier. The xylitol is usable as a special sweetener, for example.

[0038] The paper pulp or dissolving pulp obtained from the acid treatment is recovered. The pulp thus obtained is usable, when bleached, for the preparation of paper and viscose, either as such or in combination with non-acid-treated pulp.

[0039] The invention also relates to a xylose solution and to a pulp product obtained by the method and to xylose obtained from the xylose solution. The invention also relates to the use of the xylose solution thus obtained for the preparation of xylose and xylitol, and to the use of the obtained paper pulp and dissolving pulp for the preparation of paper or viscose. The invention also relates to the use of the xylose thus obtained for the preparation of flavours and/or aroma intensifiers.

[0040] The following detailed examples illustrate the present invention.

[0041] In the examples, the kappa number, viscosity, pulp yield (on dry substance of the pulp) and brightness were determined by the following methods:

Kappa number SCAN-C 1:77
Viscosity SCAN-CM 15:88
Pulp yield (on dry substance of the pulp) SCAN-C 3:78
Brightness SCAN-C 11:75.

[0042] The xylose yields (as % of dry substance of the pulp) were calculated by means of the xylose content analyzed from the cooking liquor by HPLC and by the consistency of the cooking.

Example 1. Treatment of birch sulphate pulp with formic acid.

[0043] The object was to hydrolyze xylan in birch sulphate pulp to xylose by acid treatment with a sufficient yield (>50 g xylose/1 kg starting pulp) so that the birch pulp would be suitable for dissolving pulp or paper pulp after the acid treatment. The target was a xylose yield of no less than about 5% and a viscosity of the acid-treated pulp that would be sufficient for either paper pulp or dissolving pulp.

[0044] In order for the hydrolysis to take place as efficiently as possible, the pulps were subjected to liquor exchange before the acid hydrolysis by replacing the water contained by the pulp with the acid solution to be used.

(A) Birch sulphate pulp was hydrolyzed by commercial formic acid (85%) as the acid solution. The treatment times were 20 min and 80 min. The treatment temperatures were 107°C and 115°C. The pulp used was unbleached (kappa number 18.0, brightness 30.5 and viscosity 1210 ml/g), oxygen delignified (kappa number 11.3, brightness 45.0 and viscosity 1020 ml/g) or ECF bleached (kappa number 0.6, brightness 89.0 and viscosity 890 ml/g). Table 1 shows pulp yield (%), pulp viscosity (cm^3/g), xylose yield from starting pulp (g/kg starting pulp) and theoretical yield of xylose (%).

Table 1

Formic acid treatment of birch sulphate pulp

A	B	C	D	E	F	H
7	kosa	107	20	93,2	1010	25,9
8	kosa	107	80	84,1	610	53,2
19	kosa	115	20	83,4	530	63,8
20	kosa	115	80	76,1	430	77,1
9	kosa-O	107	20	95	840	19,5
10	kosa-O	107	80	86,4	500	69,1
21	kosa-O	115	20	85,6	460	59,2
22	kosa-O	115	80	77,8	380	70,1
12	ECF	107	80	89,7	390	46,5
23	ECF	115	20	89,3	360	46,5
24	ECF	115	80	79,7	300	74,5

Specifications	
A = Test number	D = Treatment time min.
B = Pulp quality	E = Pulp yield % (after extraction)
Kosa = unbleached pulp	F = Pulp viscosity, cm^3/g
Kosa-O = oxygen delignified pulp	G = Xylose yield, g/kg starting pulp
ECF = ECF bleached pulp	H = Xylose yield on theoretical, %
C = Treatment temperature °C	

(B) Unbleached birch sulphate is hydrolyzed by an acid solution having a formic acid content of 54%. The treatment temperature is 107°C and treatment time 25 min. The viscosity of the obtained pulp is 1,000 ml/g. The carbon hydrate yield is 19.3% of the theoretical hemicellulose amount (46.2 g/kg pulp), of which 93.4% is xylose, i.e. 43.2 g/kg starting pulp (18% of the theoretical xylose yield).

(C) ECF bleached birch sulphate pulp is hydrolyzed by an acid solution having a formic acid content of 78%,

treatment temperature 107°C and treatment time 50 min. The viscosity of the obtained pulp is 600 ml/g. The carbon hydrate yield is 28% of the theoretical hemicellulose amount (67.1 g/kg), of which 92.5% is xylose, i.e. 62.1 g/kg starting pulp.

[0045] The tests conducted showed that the most preferable way to recover sugars with formic acid is from fully ECF bleached birch sulphate. At a maximum, from the pulps treated, about 185 g/kg xylose was obtained from non-bleached birch sulphate, 168 g/kg from oxygen treated birch sulphate, and about 179 g/kg from fully bleached birch sulphate.

Example 2. Treatment of birch sulphate pulp with formic acid.

[0046] Oxygen delignified birch sulphate pulp was hydrolyzed with commercial formic acid (85%) at a temperature of 107°C, the treatment times varying between 43 and 60 min. The following results were obtained:

average pulp yield 89.4% (range of variation 88.4 to 90.4%);
average pulp viscosity 650 ml/g (range of variation 600 to 710 ml/g);
average xylose yield from starting pulp 82.6 kg/1,000 kg pulp.

[0047] For the preparation of paper, the pulp was bleached with both ECF-(D-Eop-D) and TCF-(Q-P-Z/Q-P) sequences, whereby the final brightness of the ECF bleached pulp was 90.9 and that of the TCF bleached pulp 85.1.

[0048] The pulp thus obtained can be combined with non-acid-treated pulp and used for the preparation of fine paper.

Example 3. Treatment of birch sulphate pulp with formic acid, and preparation of viscose.

[0049] ECF bleached birch sulphate pulp was hydrolyzed with commercial formic acid (85%) at 107°C for 50 minutes, whereby the following results were obtained:

pulp yield 93.9%
pulp viscosity 470 ml/g
xylose yield from starting pulp 78.8 kg/1,000 kg.

[0050] The pulp thus treated was washed and used as such for the preparation of viscose. The viscose was prepared as follows:

[0051] Mercerization was carried out as slurry mercerization by elutriating chemical pulp in 18.0% (w/w) NaOH solution at 50°C for 20 minutes (doses 42.5 g chemical pulp/1 litre NaOH solution).

[0052] After mercerization, the alkali cellulose was filtered from the slurry so as to form a cake, which was hydraulically pressed to a suitable dry substance content. The obtained contents of the alkali cellulose were: 32.14% alpha cellulose and 15.11 % NaOH. These values corresponded to normal values. The pressed cake was torn in a mixer so as to obtain flaky pulp, which was prematured for 22 hours at 34°C to obtain a suitable DP level.

[0053] To the prematured alkali cellulose, 35% carbon bisulphide of the alpha cellulose content of the alkali cellulose was added under reduced pressure. The sulphuring was carried out at 32°C during 1 h 15 min.

[0054] The cellulose xanthate generated in the sulphuring was dissolved with a dilute NaOH solution during 4 hours at 20°C. The water and NaOH amounts in the NaOH solution were adjusted such that the viscose contents obtained were 6% NaOH and 8% alpha cellulose.

[0055] The dissolved viscose was post-matured for 24 hours at 20°C, whereupon the ball viscosity and the drainability were measured from the viscose.

[0056] The ball viscosity of the thus obtained viscose (i.e. the time required for a steel ball having a 3-mm diameter to sink a 20-cm distance in a viscose solution) was 37.5 seconds, i.e. the viscose was relatively fluid.

Example 4. Treatment of birch sulphate pulp with bisulphite solution.

[0057] Unbleached birch sulphate pulp was hydrolyzed with an aqueous solution of bisulphite. The purpose was to hydrolyze xylan from the pulp into xylose with a good yield and simultaneously retain the paper production properties of the pulp as good as possible. The target yield of xylose was 5% or more. The viscosity of the pulp was used as the measure of the paper production properties, and the target viscosity was set to be not less than 450 ml/g.

[0058] The treated pulp was then bleached to the target brightness of 85 ISO or 90 ISO.

[0059] The raw material used was conventional birch sulphate pulp. The pulp had the following characteristics:

total yield, %	51.8
screening yield, %	50.4
reject, %	1.4
kappa number	20.6
viscosity, ml/g	1350
brightness, ISO	26.9

[0060] A sulphite cooking liquor was used in the hydrolysis. The total SO_2 content of the solution was 3%, of which 10% was bound SO_2. The binding cation was Na+.

[0061] The hydrolysis was carried out in an acid-resistant steel autoclave having a volume of 1 dm^3. The pulp and the sulphite cooking liquor were heated in an air bath starting from room temperature up to the final treatment temperature (130 or 140°C), the duration of the reaction being 1 to 3 h.

[0062] After the hydrolysis, the autoclave was cooled to room temperature. The hydrolysis solution was filtered from the mixture with a vacuum filter, and the xylose content of the solution was measured.

[0063] The pulp was washed with water. The washing water was filtered from the mixture and the pulp was centrifuged and homogenized. The yield, viscosity, kappa number and brightness of the pulp were measured. The results are shown in Table 2.

Table 2

Treatment of birch sulphate pulp with bisulphite								
Test	**Temperature, °C**	**Time, min**	**Bound SO_2, %**	**Pulp yield, %**	**Xylose yield, %**	**Viscosity, ml/g**	**Kappa number**	**Brightness %**
8	140	120	10	87,3		570		48,5
9	140	180	10	84,7		490		47,3
10	130	60	10	92,1		810		46,4
11	130	120	10	90,4	4,4	670	9,7	46,8
12	130	180	0	89,0		620	9,2	47,7
15	130	180	10	89,6	5,3	630	8,9	47,2
16	130	120	10	90,7		710	9,2	46,5

[0064] The yield values of the pulps show that sufficient amounts of xylose were separated from the pulp, i.e. the xylose yields approximately corresponded to the target values, as did the viscosity values. in tests 11 and 15, xylose yields (as % of dry substance of the pulp) were also separately determined. The results confirmed that the xylose yields approximately correspond to the target values.

[0065] The pulps obtained from tests 15 and 16 were combined and bleached with the sequence O_p - D - P to the target brightness.

[0066] In addition to birch sulphate pulp, the method can be applied to other hardwood pulp prepared by alkaline or neutral cooking or to pulp prepared from herbaceous plants. In the post hydrolysis, other organic or inorganic acids besides formic acid and a bisulphite solution may also be used.

Claims

1. A method of preparing chemical pulp and a xylose solution by the use of alkaline or neutral cooking and a post-hydrolysis of the pulp, **characterized in that** the post-hydrolysis is performed directly on the pulp by the use of an acid by post-hydrolyzing the pulp with an acid until a xylose yield of no less than 5% is obtained, while the viscosity of the pulp remains at a value of no less than 300 ml/g.

2. A method as claimed in claim 1, **characterized in that** the pulp is post-hydrolyzed with an acid until a xylose yield of no less than 10% is obtained, while the viscosity of the pulp remains at a value of no less than 450 ml/g.

3. A method as claimed in claim 1 or 2, **characterized in that** the acid treatment is carried out with formic acid.

4. A method as claimed in claim 3, **characterized in that** the content of the acid solution is within the range 50 to 100%, preferably 75 to 90%.

5. A method as claimed in claim 3 or 4, **characterized in that** the acid treatment temperature is between 90 and 130°C, preferably between 100 and 120°C.

6. A method as claimed in any one of claims 3 to 5, **characterized in that** the duration of the acid treatment is between 15 min and 4 h, preferably between 20 min and 1.5 h.

7. A method as claimed in claim 1, **characterized in that** the acid treatment is performed with a bisulphite solution.

8. A method as claimed in claim 7, **characterized in that** the SO_2 content of the bisulphite solution is within the range from about 1 to about 5%, preferably about 3%.

9. A method as claimed in claim 8, **characterized in that** the amount of bound SO_2 is about 10%.

10. A method as claimed in any one of claims 7 to 9, **characterized in that** the acid treatment temperature is about 110 to 150°C, preferably about 125 to 145°C.

11. A method as claimed in any one of claims 7 to 10, **characterized in that** the duration of the acid treatment is 1 to 3 h.

12. A method as claimed in any one of the preceding claims, **characterized in that** the acid treatment is performed after cooking.

13. A method as claimed in any one of claims 1 to 11, **characterized in that** the acid treatment is performed after oxygen delignification.

14. A method as claimed in any one of claims 1 to 11, **characterized in that** the acid treatment is performed after bleaching.

15. A method as claimed in any one of the preceding claims, **characterized in that** the cooking is performed by the sulphate method, whereby the post hydrolysis is carried out directly on the pulp by the use of an acid.

16. A method as claimed in any one of claims 1 to 15, **characterized in that** birch is used as the raw material in the cooking.

17. A method as claimed in any one of the preceding claims, **characterized in that** after the acid treatment the obtained xylose solution and the chemical pulp are separated.

18. A method as claimed in claim 17, **characterized in that** the acid used in the acid treatment is separated from the obtained xylose solution.

19. A method as claimed in claim 18, **characterized in that** the separated acid is recycled and reused in the hydrolysis.

20. A method as claimed in any one of claims 1 to 17, **characterized in that** the obtained chemical pulp is recovered.

21. A method as claimed in claim 20, **characterized in that** the obtained chemical pulp is mixed with non-acid-treated pulp.

22. The use of the xylose solution obtainable in accordance with any of claims 1 to 21 for the preparation of xylitol, flavours and/or aroma intensifiers.

23. The use of the chemical pulp obtainable in accordance with any one of claims 1 to 21 for the preparation of paper or viscose.

Patentansprüche

1. Verfahren zur Herstellung eines chemischen Zellstoffs und einer Xylose-Lösung durch die Anwendung eines alkalischen oder neutralen Kochens und einer Nachhydrolyse des Zellstoffs, **dadurch gekennzeichnet, daß** die Nachhydrolyse direkt an dem Zellstoff durch Verwendung einer Säure durchgeführt wird, indem der Zellstoff mit einer Säure nachhydrolysiert wird, bis eine Xylose-Ausbeute von nicht weniger als 5 % erreicht ist, während die Viskosität des Zellstoffs bei einem Wert von nicht weniger als 300 ml/g bleibt.

2. Verfahren nach Anspruch 1, **dadurch gekennzeichnet, daß** der Zellstoff mit einer Säure nachhydrolysiert wird, bis eine Xylose-Ausbeute von nicht weniger als 10 % erreicht ist, während die Viskosität des Zellstoffs bei einem Wert von nicht weniger als 450 ml/g bleibt.

3. Verfahren nach Anspruch 1 oder Anspruch 2, **dadurch gekennzeichnet, daß** die Säurebehandlung mit Ameisensäure durchgeführt wird.

4. Verfahren nach Anspruch 3, **dadurch gekennzeichnet, daß** der Gehalt der Säurelösung im Bereich von 50 bis 100 %, vorzugsweise 75 bis 90 % liegt.

5. Verfahren nach Anspruch 3 oder Anspruch 4, **dadurch gekennzeichnet, daß** die Säurebehandlungstemperatur zwischen 90 und 130°C, vorzugsweise zwischen 100 und 120°C liegt.

6. Verfahren nach einem der Ansprüche 3 bis 5, **dadurch gekennzeichnet, daß** die Dauer der Säurebehandlung zwischen 15 min und 4 h, vorzugsweise zwischen 20 min und 1,5 h liegt.

7. Verfahren nach Anspruch 1, **dadurch gekennzeichnet, daß** die Säurebehandlung mit einer Bisulfit-Lösung durchgeführt wird.

8. Verfahren nach Anspruch 7, **dadurch gekennzeichnet, daß** der SO_2-Gehalt der Bisulfit-Lösung im Bereich von etwa 1 bis etwa 5 % liegt, vorzugsweise etwa 3 % ist.

9. Verfahren nach Anspruch 8, **dadurch gekennzeichnet, daß** die Menge an gebundenem SO_2 etwa 10 % ist.

10. Verfahren nach einem der Ansprüche 7 bis 9, **dadurch gekennzeichnet, daß** die Säurebehandlungstemperatur etwa 110 bis 150°C, vorzugsweise etwa 125 bis 145°C ist.

11. Verfahren nach einem der Ansprüche 7 bis 10, **dadurch gekennzeichnet, daß** die Dauer der Säurebehandlung 1 bis 3 h ist.

12. Verfahren nach einem der vorangehenden Ansprüche, **dadurch gekennzeichnet, daß** die Säurebehandlung nach einem Kochen durchgeführt wird.

13. Verfahren nach einem der Ansprüche 1 bis 11, **dadurch gekennzeichnet, daß** die Säurebehandlung nach einer Sauerstoffdelignifizierung durchgeführt wird.

14. Verfahren nach einem der Ansprüche 1 bis 11, **dadurch gekennzeichnet, daß** die Säurebehandlung nach Bleichen durchgeführt wird.

15. Verfahren nach einem der vorangehenden Ansprüche, **dadurch gekennzeichnet, daß** das Kochen durch das Sulfat-Verfahren durchgeführt wird, wodurch die Nachhydrolyse direkt durch Verwendung einer Säure an dem Zellstoff durchgeführt wird.

16. Verfahren nach einem der Ansprüche 1 bis 15, **dadurch gekennzeichnet, daß** Birke aus Ausgangsmaterial beim Kochen eingesetzt wird.

17. Verfahren nach einem der vorangehenden Ansprüche, **dadurch gekennzeichnet, daß** nach der Säurebehandlung die erhaltene Xylose-Lösung und der chemische Zellstoff getrennt werden.

18. Verfahren nach Anspruch 17, **dadurch gekennzeichnet, daß** die bei der Säurebehandlung verwendete Säure

von der erhaltenen Xylose-Lösung abgetrennt wird.

19. Verfahren nach Anspruch 18, **dadurch gekennzeichnet, daß** die abgetrennte Säure recyclet und bei der Hydrolyse wieder verwendet wird.

20. Verfahren nach einem der Ansprüche 1 bis 17, **dadurch gekennzeichnet, daß** der erhaltene chemische Zellstoff isoliert wird.

21. Verfahren nach Anspruch 20, **dadurch gekennzeichnet, daß** der erhaltene chemische Zellstoff mit nichtsäurebehandeltem Zellstoff vermischt wird.

22. Verwendung der Xylose-Lösung, die gemäß einem der Ansprüche 1 bis 21 erhältlich ist, zur Herstellung von Xylit, Aromastoffen und/oder Aromaintensivierungsmitteln.

23. Verwendung des chemischen Zellstoffs, der nach einem der Ansprüche 1 bis 21 erhältlich ist, zur Herstellung von Papier oder Viskose.

Revendications

1. Procédé permettant de préparer une pâte chimique et une solution de xylose, par cuisson alcaline ou neutre et post-hydrolyse de la pâte, caractérisé en que l'on soumet directement la pâte à une post-hydrolyse effectuée à l'aide d'un acide, en poussant la post-hydrolyse de la pâte par un acide jusqu'à obtenir un rendement en xylose d'au moins 5 %, alors que la visco-sité de la pâte reste au moins égale à 300 ml/g.

2. Procédé conforme à la revendication 1, **caractérisé en ce que** l'on pousse la post-hydrolyse de la pâte par un acide jusqu'à obtenir un rendement en xylose d'au moins 10 %, alors que la viscosité de la pâte reste au moins égale à 450 ml/g.

3. Procédé conforme à la revendication 1 ou 2, **caractérisé en ce que** le traitement à l'acide est effectué avec de l'acide formique.

4. Procédé conforme à la revendication 3, **caractérisé en ce que** la teneur de la solution d'acide vaut de 50 à 100 %, et de préférence de 75 à 90 %.

5. Procédé conforme à la revendication 3 ou 4, **caractérisé en ce que** le traitement à l'acide est effectué à une température de 90 à 130 °C, et de préférence de 100 à 120 °C.

6. Procédé conforme à l'une des revendications 3 à 5, **caractérisé en ce que** le traitement à l'acide dure de 15 minutes à 4 heures, et de préférence de 20 minutes à 1,5 heure.

7. Procédé conforme à la revendication 1, **caractérisé en ce que** le traitement à l'acide est effectué à l'aide d'une solution de bisulfite.

8. Procédé conforme à la revendication 7, **caractérisé en ce que** la teneur en SO_2 de la solution de bisulfite vaut à peu près de 1 à 5 %, et de préférence environ 3 %.

9. Procédé conforme à la revendication 8, **caractérisé en ce que** la quantité de SO_2 lié vaut à peu près 10 %,

10. Procédé conforme à l'une des revendications 7 à 9, **caractérisé en ce que** le traitement à l'acide est effectué à une température d'à peu près 110 à 150 °C, et de préférence d'à peu près 125 à 145 °C.

11. Procédé conforme à l'une des revendications 7 à 10, **caractérisé en ce que** le traitement à l'acide dure de 1 à 3 heures.

12. Procédé conforme à l'une des revendications précédentes, **caractérisé en ce que** le traitement à l'acide est effectué après cuisson.

13. Procédé conforme à l'une des revendications 1 à 11, **caractérisé en ce que** le traitement à l'acide est effectué après délignification à l'oxygène.

14. Procédé conforme à l'une des revendications 1 à 11, **caractérisé en ce que** le traitement à l'acide est effectué après blanchiment.

15. Procédé conforme à l'une des revendications précédentes, **caractérisé en ce que** la cuisson est effectuée selon le procédé au sulfate, la pâte étant directement soumise à la post-hydrolyse effectuée à l'aide d'un acide.

16. Procédé conforme à l'une des revendications 1 à 15, **caractérisé en ce qu'**on utilise du bouleau en tant que matière première pour la cuisson.

17. Procédé conforme à l'une des revendications précédentes, **caractérisé en ce que**, après le traitement à l'acide, on sépare la solution de xylose obtenue et la pâte chimique.

18. Procédé conforme à la revendication 17, **caractérisé en ce qu'**on sépare l'acide employé dans le traitement à l'acide d'avec la solution de xylose obtenue.

19. Procédé conforme à la revendication 18, **caractérisé en ce que** l'acide séparé est recyclé et réutilisé dans l'hydrolyse.

20. Procédé conforme à l'une des revendications 1 à 17, **caractérisé en ce qu'**on récupère la pâte chimique obtenue.

21. Procédé conforme à la revendication 20, **caractérisé en ce qu'**on mélange la pâte chimique obtenue avec de la pâte qui n'a pas subi de traitement à l'acide.

22. Emploi d'une solution de xylose obtenue conformément à l'une des revendications 1 à 21 dans la fabrication de xylitol, d'arômes et/ou d'intensificateurs d'arômes.

23. Emploi d'une pâte chimique obtenue conformément à l'une des revendications 1 à 21 dans la fabrication de papier ou de viscose.